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13. ABSTRACT *(Maximum 200 words)*

We report continuing results pertaining to the above referenced grant, DAAG55-98-1-0511. As detailed herein, we have successfully synthesized a number of metal cluster core-based star architectures. These architectures are designed so that their encapsulating properties can be directly compared with those of dendrimers prepared previously in our research group. We are now embarking on the measurement of the electrochemical properties of these star molecules and will then publish these results as a direct comparison of star and hyperbranched architectures as regards their encapsulating properties. Encapsulation is a critical behavior in the design of nanoscale electronic elements, nanoscale sensors, etc.

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(1) LIST OF MANUSCRIPTS

None submitted. A manuscript describing these results is in preparation

(2) SCIENTIFIC PERSONEL

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(3) INVENTIONS

None

(4) SCIENTIFIC PROGRESS AND ACCOMPLISHMENTS

We report continuing results pertaining to our proposal, "Comparison of the Organization of Linear and Hyperbranched Structures Using Metal-Cluster Core Dendritic and Star Architectures...", DAAG55-98-1-0511. As detailed below, we have successfully synthesized a number of metal cluster core-based star architectures. These architectures are designed so that their encapsulating properties can be directly compared with those of dendrimers prepared previously in our research group. We are now embarking on the measurement of the electrochemical properties of these star molecules and will then publish these results as a direct comparison of star and hyperbranched architectures as regards their encapsulating properties. Encapsulation is a critical behavior in the design of nanoscale electronic elements, nanoscale sensors, etc.

A summary of accomplishments in this area are:

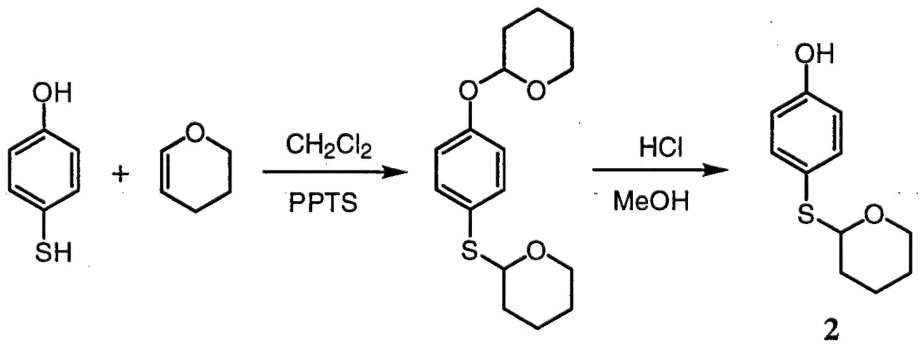
A central issue in the use of dendrimers in applications is whether or not their unique architecture is really ideal or even necessary. Could a much simpler linear polymer serve just as well as a hyperbranched polymer or dendrimer? This question seems fundamental in designing a commercial process as dendrimers are hard to make (requiring stepwise synthesis and chromatography) and also can be hard to characterize (for example, molecular weight determinations using size exclusion chromatography are generally invalid for dendrimers due to their very different size/molecular weight profile compared to linear polymer standards). In contrast, much information is available for linear polymers and they are almost universally easier to prepare and characterize. Many of the justifications for the use of dendrimers in applications have revolved around their size, monodispersity, shape, and functional group accessibility. It is likely that dendrimers offer

advantages in these areas compared to conventional linear macromolecules. However, with little to no information available that illustrates the effects of these structural features, it is surprising that dendrimers were initially employed in these applications. It is likewise valid to ask whether other, simpler macromolecules can and perhaps ultimately will take their place. **Thus, if the real use of dendrimers in applications is to be sustained and supported, it is worthy to illustrate features of dendrimers that are inaccessible using other, more straightforward macromolecular architectures.**

To these ends, we have prepared molecules of the form $[Fe_4S_4(S\text{-}Poly)_4]^{2-}$ to compare with the molecules previously reported by us of the form $[Fe_4S_4(S\text{-}Dend)_4]^{2-}$. Here, the "Poly" moiety is ideally a well-defined linear chain with a molecular weight in the 100-5000 range, a polydispersity that is as low as possible, and a way of terminating the chain in an aromatic thiol group so it can be attached to an iron-sulfur cluster.

We have devised a protocol for substituting polymers with aromatic thiol groups so that star-cluster polymers can be synthesized. Although it was envisioned that simple thiol protection group chemistry could be utilized, it was discovered that a number of thiol protection groups are generally incompatible with the types of polymer linking chemistry that would be most profitable in this type of endeavor. Thus, we devised the molecule **2** shown in Scheme I below as an ideal protected aromatic thiol that was compatible with the type of functionalization chemistry we ultimately wished to pursue.

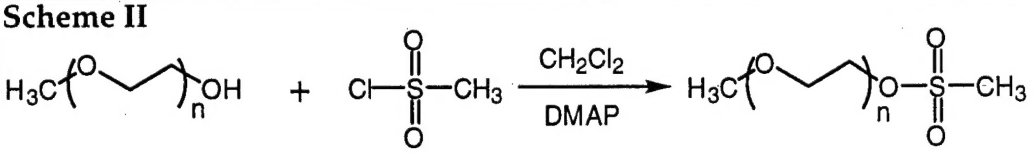
Scheme I



1 **2**

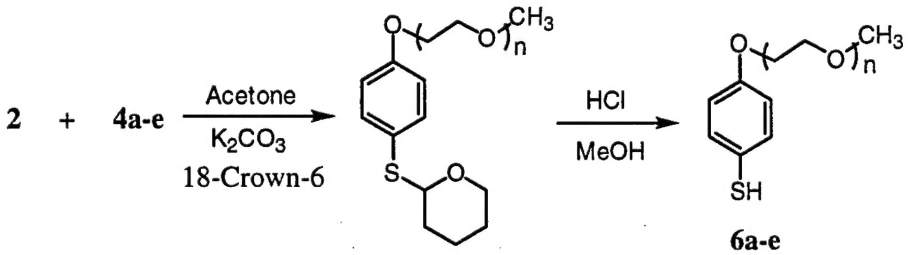
The use of poly(ethylene glycol) (PEG) as the polymer was obvious given that it can be obtained in a variety of molecular weights with specific functional groups on each end. We obtained PEG with molecular weights shown in Scheme II and functionalized it so that it could be coupled to unit **2** as shown in Scheme II. Appended to this report is a series of mass spectra that illustrate complete functionalization for each of the three steps shown in this scheme.

Scheme II



3a-e **4a-e**

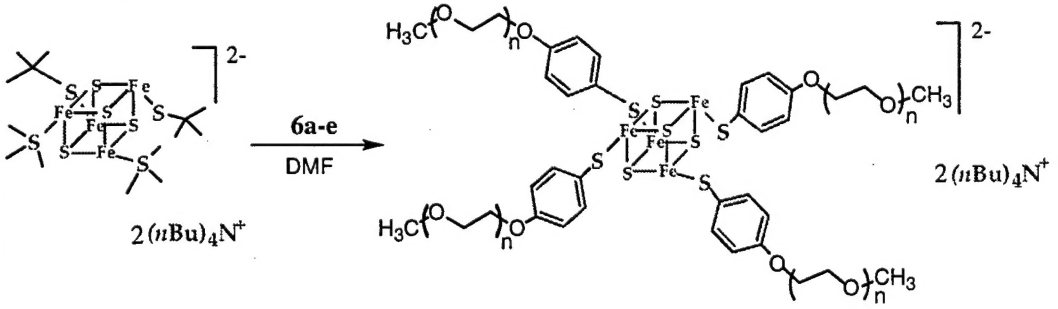
a: n = 1
b: Average M_n ca. = 350;
c: Average M_n ca. = 750;
d: Average M_n ca. = 2000;
e: Average M_n ca. = 5000.



5a-e **6a-e**

With a series of thiol-functionalized polymers in hand, we were able to make the iron-sulfur clusters as shown in Scheme III. Success in this reaction was easily illustrated with NMR in which a shift in the peaks due to aromatic ring protons from ca. 7 ppm in the starting material to 6 ppm and 8 ppm in the product was observed (Figure 1).

Scheme III



Clusters 7a-e

In the next 1-2 week period, the electrochemical behavior of these molecules will be measured and compared with dendrimer analogues. A manuscript on this work will then be submitted.

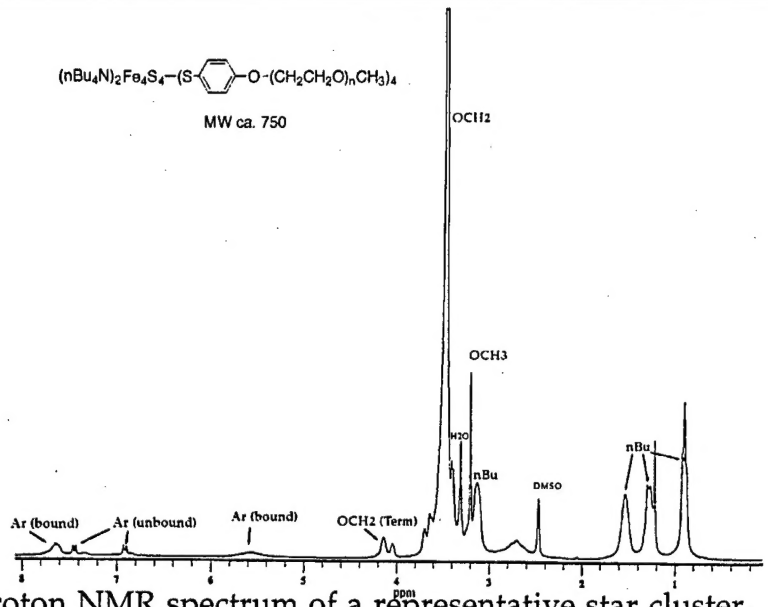


Figure 1. Proton NMR spectrum of a representative star cluster.

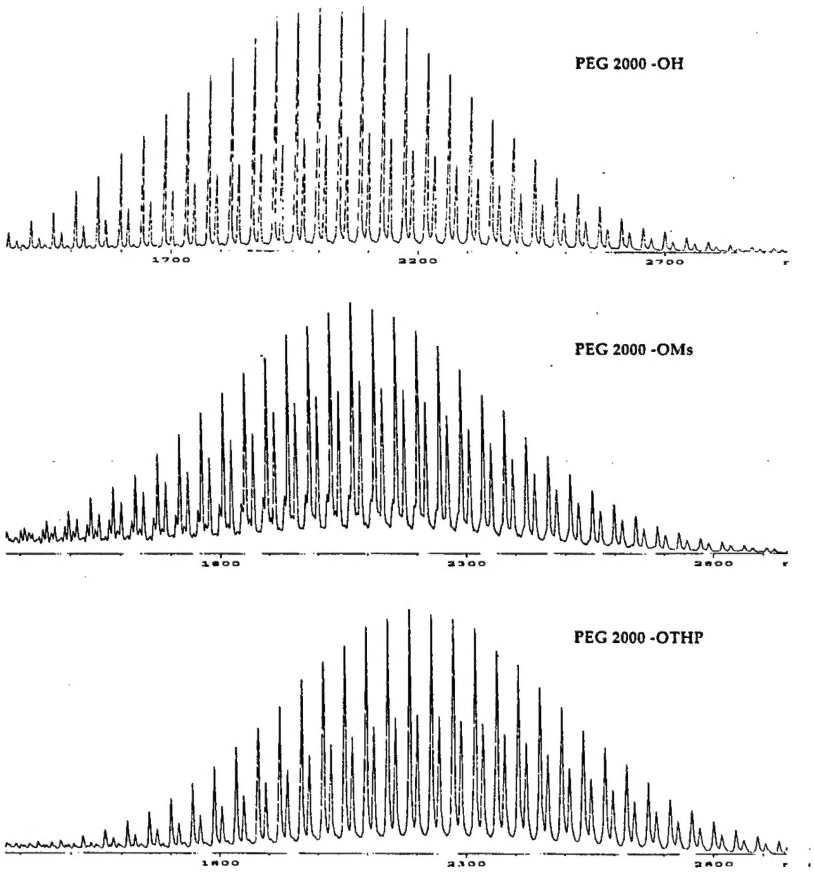


Figure 2. Mass spectra illustrating the efficiacy of the polymer functionalization reactions employed

Experimental Section

General. The reactions to synthesize all clusters were carried out under a partial vaccum. All other reactions were run under a nitrogen atmosphere. ^{1}H NMR and ^{13}C NMR spectra were recorded on a GE 300 MHz and 75 MHz FT-NMR spectrometer respectively. The UV-Vis absorption spectra were obtained on a Hewlett Packard 8452A spectrophotometer. Matrix assisted laser deposition ionization time of flight (MALDI-TOF) mass spectra were obtained using a Bruker Proflex III instrument.

Materials. Ethyl ether and tetrahydrofuran (THF) were distilled from sodium benzophenone ketyl. Dichloromathane was distilled from potassium hydroxide. The starting iron-sulfur cluster $[Fe_4S_4(S\text{-}tBu)_4]^{2-}\cdot 2(nBu_4N^+)$ was prepared as described in the literature (Christou, G.; Garner, C.D. J.C.S. Dalton, 1979, 1093). All other starting materials purchased from Aldrich and TCI were used without purification unless otherwise noted.

Synthesis. Compound 1: To a mixture of 4-hydroxythiophenol (12.62 g, 100 mmol) and pyridinium p-toluene sulfonate (PPTS, 2.51 g, 10 mmol) in 150 ml of distilled dichloromethane, 3,4-dihydro-2H-pyran (29.44 g, 350 mmol) was added dropwise. The resulting mixture was stirred under overnight, and washed with 10% sodium hydroxide aqueous solution (3 × 50 ml). The organic layer was dried over MgSO4. The solvent was removed from the filtrate under reduced pressure, yield: 75%. ^{1}H NMR (acetone-d_6, ppm) δ 1.58 – 1.95 (m, 8H, CH_2), 3.50 (m, 2H, OCH_2), 3.80 (m, 2H, OCH_2), 5.06 (t, 1H, OCH, $J_{H\text{-}H}$ = 5.1 Hz), 5.40 (t, 1H, SCH, $J_{H\text{-}H}$ = 3.7 Hz), 6.97 (m, 2H, C_6H_4), 7.38 (m, 2H, C_6H_4). ^{13}C NMR (acetone-d_6, ppm) δ 19.29, 21.94, 25.66, 26.05, 26.11, 32.02, 62.20, 64.30, 86.34, 96.78, 117.49, 127.18, 134.32, 157.46. HRMS (EI, m/z): 294.1290 Found 294.1290, Δ < 0.1 ppm. Anal calcd for $C_{16}H_{22}O_3S$ (294.42): C, 65.27%; H, 7.53%; S, 10.89% Found C, 65.17%; H, 7.56%; S, 10.75%.

Compound 2: To a solution of in 200 ml of methanol at 0 °C, 3 ml of 4M HCl solution was added dropwise over 20 min with stirring. The resulting mixture was further stirred for 2 h. 200 ml of ethyl ether was added, then washed with saturated sodium bicarbonate aqueous solution (2 × 50 ml). The organic layer was dried over $MgSO_4$. The solvent was removed from the filtrate under reduced pressure. The purification by recrystallization from hexanes gave a white solid (yield: 95%). ^{1}H NMR (acetone-d_6, ppm) δ 1.58 – 1.95 (m, 4H, CH_2), 3.42 (m, 2H, OCH_2), 4.04 (m, 2H, OCH_2), 4.97 (m, 1H, SCH), 6.77 (m, 2H, C_6H_4), 7.30 (m, 2H, C_6H_4), 8.44 (s, 1H, OH). ^{13}C NMR (acetone-d_6, ppm) δ 22.65, 26.14, 31.99, 64.40, 86.60, 116.39, 124.50, 135.22, 157.91. HRMS (EI, m/z): 210.0715 Found 210.0718, Δ = 1.6 ppm. Anal calcd for $C_{11}H_{14}O_2S$ (210.07): C, 62.83%; H, 6.71%; S, 15.41% Found C, 62.58%; H, 6.67%; S, 15.41%.

A general procedure for the synthesis of compounds 4a-e is outlined below. Compound 4a: To a mixture of 2-methoxyethanol (2.0 g, 26.3 mmol), triethylamine (10.7 g, 105 mmol) and 4-dimethylaminopyridine (DMAP, 64 mg, 0.53 mmol) in dichloromathane (100 ml) at 0 °C, methanesulfonyl chloride (9.0 g, 86.9 mmol) was added dropwise with stirring. After completion of the addition, the resulting mixture was warmed to room temperature, and further stirred overnight. The organic layer was washed with 1M HCl solution (2 × 50 ml) and water (2 × 50 ml), and then dried over Na_2SO_4. The solvent was removed from the

filtrate under reduced pressure, yield: 90%. ^{1}H NMR ($CDCl_3$, ppm) δ 3.04 (s, 3H, SCH_3), 3.39 (s, 3H, OCH_3), 3.65 (t, 2H, OCH_2, J_{H-H} = 4.4 Hz), 4.53 (t, 2H, OCH_2, J_{H-H} = 4.4 Hz). ^{13}C NMR ($CDCl_3$, ppm) δ 38.26, 53.25, 59.59, 69.57, 70.02, 71.06, 71.12, 72.48. HRMS (CI, m/z): 155.0378 Found 155.0376, Δ = 1.3 ppm. Anal calcd for $C_4H_{10}O_4S$ (154.19): C, 31.16%; H, 6.54%; S, 20.80%. Found C, 31.32%; H, 6.66%; S, 20.92%.

Compound 4b: yield: 92%. ^{1}H NMR ($CDCl_3$, ppm) δ 2.98 (s, 3H, SCH_3), 3.26 (s, 3H, OCH_3), 3.53 (m, 2H, CH_2OMe), 3.64 (m, OCH_2), 3.67 (m, 2H, CH_2CH_2OMe), 4.26 (m, 2H, CH_2OMe). ^{13}C NMR ($CDCl_3$, ppm) δ 38.42, 53.28, 59.75, 69.70, 70.02, 71.25. MS (FAB): M_w = 468.5, M_n = 446.5, M_w/M_n = 1.05.

Compound 4c: yield: 95%. ^{1}H NMR ($CDCl_3$, ppm) δ 2.93 (s, 3H, SCH_3), 3.21 (s, 3H, OCH_3), 3.37 (m, 2H, CH_2OMe), 3.49 (m, OCH_2), 3.60 (m, 2H, CH_2CH_2OMe), 4.22 (m, 2H, CH_2OMe). ^{13}C NMR ($CDCl_3$, ppm) δ 38.20, 59.52, 69.54, 69.96, 71.06, 72.45. MS (FAB): M_w = 838.3, M_n = 819.9, M_w/M_n = 1.02.

Compound 4d: yield: 95%. ^{1}H NMR ($CDCl_3$, ppm) δ 3.05 (s, 3H, SCH_3), 3.32 (s, 3H, OCH_3), 3.50 (m, 2H, CH_2OMe), 3.58 (m, OCH_2), 3.71 (m, 2H, OCH_2), 4.33 (m, 2H, CH_2OMe). ^{13}C NMR ($CDCl_3$, ppm) δ 53.28, 59.77, 69.73, 70.02, 71.28, 72.64. MS (MALDI-TOF, dithranol): M_w = 2180, M_n = 2000, M_w/M_n = 1.05.

Compound 4e: yield: 97%. ^{1}H NMR ($CDCl_3$, ppm) δ 2.99 (s, 3H, SCH_3), 3.28 (s, 3H, OCH_3), 3.55 (m, 2H, CH_2OMe), 3.77 (m, OCH_2), 4.24 (m, 2H, CH_2OMe). ^{13}C NMR ($CDCl_3$, ppm) δ 69.62, 69.98, 71.15, 72.51. MS (MALDI-TOF, dithranol): M_w = 2180, M_n = 2000, M_w/M_n = 1.05.

A general procedure for the synthesis of compounds 5a-e is outlined below. Compound 5a: A mixture of 4a (3.0 g, 5.66 mmol), 2 (1.1 g, 7.3 mmol), potassium carbonate (4.7 g, 44 mmol) and 18-crown-6 (0.30 g, 1.13 mmol) in 100 ml of acetone was refluxed under nitrogen for 70 h. After cooled to room temperature, the solvent was removed under reduced pressure. The residue was dissolved into 50 ml of ethyl acetate, and the organic layer was washed with saturated NH_4Cl solution (2 × 30 ml) and sodium chloride solution (2 × 30 ml), and then dried over Na_2SO_4. The solvent was removed from the filtrate under reduced pressure (yield: 85%). ^{1}H NMR ($CDCl_3$, ppm) δ 1.55 – 1.96 (m, 6H, OCH_2), 3.36 (s, 3H, OCH_3), 3.67 (s, 2H, OCH_2), 4.10 (m, 2H, OCH_2), 5.04 (t, 1H, SCH, J_{H-H} = 3.7 Hz), 6.88 (m, 2H, C_6H_4), 7.41 (m, 2H, C_6H_4). ^{13}C (acetone-d_6, ppm) δ 21.97, 26.14, 32.02, 58.68, 64.37, 67.95, 71.38, 86.44, 115.55, 126.18, 134.71. HRMS (EI, m/z): 268.1133 Found 268.1132, Δ = 0.5 ppm. Anal calcd for $C_{14}H_{20}O_3S$ (268.38): C, 62.66%; H, 7.51%; S, 11.95%. Found C, 62.77%; H, 7.63%; S, 12.07%.

Compound 5b: 82%. ^{1}H NMR (acetone-d_6, ppm) δ 1.56 – 1.96 (m, 4H, CH_2), 3.25 (s, 3H, CH_3), 3.43 (m, 2H, OCH_2), 3.57 (m, OCH_2), 3.78 (m, 2H, OCH_2), 4.12 (m, 2H, OCH_2), 5.07 (m, 1H, SCH), 6.89 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.38 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz). ^{13}C NMR (acetone-d_6, ppm) δ 21.97, 26.11, 32.02, 58.55, 64.37, 68.21, 70.02, 70.83, 70.99, 71.19, 72.41, 86.44, 115.62, 126.15, 134.71, 159.40. MS (FAB): M_w = 430, M_n = 400, M_w/M_n = 1.05.

Compound 5c: yield: 80%. ^{1}H NMR (acetone-d_6, ppm) δ 1.56 – 1.96 (m, 4H, CH_2), 3.26 (s, 3H, CH_3), 3.43 (m, 2H, OCH_2), 3.57 (m, OCH_2), 3.62 (m, 2H, OCH_2), 3.78 (m,

2H, OCH_2), 4.12 (m, 2H, OCH_2), 5.06 (m, 1H, SCH), 6.89 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.38 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz). ^{13}C NMR (acetone-d_6, ppm) δ 21.97, 26.11, 32.02, 58.58, 64.37, 68.21, 70.02, 70.83, 71.02, 71.19, 71.31, 72.41, 86.44, 115.65, 126.12, 134.72, 159.40. MS (FAB): M_w = 430, M_n = 400, M_w/M_n = 1.05.

Compound 5d: 75%. ^1H NMR (acetone-d_6, ppm) δ 1.54 – 1.96 (m, 4H, OCH_2), 2.03 (m, 2H, OCH_2), 3.27 (s, 3H, CH_3), 3.35 (m, 2H, OCH_2), 3.58 (m, OCH_2), 3.64 (m, 2H, OCH_2), 3.77 (m, 2H, OCH_2), 4.14 (m, 2H, OCH_2), 5.07 (m, 1H, SCH), 6.89 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.38 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz). ^{13}C NMR (acetone-d_6, ppm) δ 21.97, 26.14, 32.02, 58.62, 64.37, 68.21, 70.06, 70.70, 70.86, 71.02, 71.19, 71.32, 72.45, 86.44, 115.68, 126.12, 134.71, 159.40. MS (MALDI-TOF, dithranol): M_w = 2180, M_n = 2000, M_w/M_n = 1.05.

Compound 5e: 78%. ^1H NMR (acetone-d_6, ppm) δ 1.53 – 1.94 (m, 4H, OCH_2), 3.26 (s, 3H, CH_3), 3.34 (m, 2H, OCH_2), 3.57 (m, OCH_2), 3.62 (m, 2H, OCH_2), 3.78 (m, 2H, OCH_2), 4.12 (m, 2H, OCH_2), 5.05 (m, 1H, SCH), 6.89 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.38 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz). ^{13}C NMR (acetone-d_6, ppm) δ 21.97, 26.14, 32.38, 64.67, 68.21, 70.05, 70.66, 70.73, 70.83, 71.02, 71.19, 105.02, 115.65, 134.71. MS (MALDI-TOF, dithranol): M_w = 2180, M_n = 2000, M_w/M_n = 1.05.

A general procedure for the synthesis of compounds 6a-e is outlined below. Compound 6a: To a solution of 5a (532mg, 2 mmol) in 30 ml of methanol at 0 °C, 4 ml of 3M HCl aqueous solution was added dropwise. After completion of the addition, sodium nitrate (210 mg, 3 mmol) was added, the reaction mixture turuned into green color immediately, and the color disappeared after 1 h. After then, the mixture was warmed to room temperature, and further stirred under nitrogen for another 6 h. Ethyl acetate was added to extract the product, and the organic layer was washed with water (2 × 50 ml). The organic layer was dried over $MgSO_4$. The solvent was removed from the filtrate under reduced pressure (yield: 96%). MS (EI, m/z): 184 found 184.

Compound 6c: 95%. ^1H NMR (acetone-d_6, ppm) δ 2.78(s, 1H, SH), 3.26 (s, 3H, CH_3), 3.34 – 3.65 (m, OCH_2), 3.50 (s, OCH_2), 3.81 (m, CH_2), 4.15 (m, OCH_2), 6.95 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.42 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz).

Compound 6d: 97%. ^1H NMR (acetone-d_6, ppm) δ 2.81 (s, 1H, SH), 3.29 (s, 3H, CH_3), 3.34 (m, 2H, OCH_2), 3.47 (s, OCH_2), 3.58 (m, 2H, OCH_2), 3.64 (m, 2H, OCH_2), 3.73 (m, 2H, OCH_2), 3.82 (m, 2H, OCH_2), 4.15 (m, 2H, OCH_2), 6.95 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.42 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz). ^{13}C NMR (acetone-d_6, ppm) δ 58.58, 68.41, 70.02, 70.86, 71.06, 71.22, 71.35, 72.45, 116.13, 133.10, 160.30. MS (MALDI-TOF, dithranol): M_w = 2180, M_n = 2000, M_w/M_n = 1.05.

Compound 6e: 95%. ^1H NMR (acetone-d_6, ppm) δ 2.82 (m, OCH_2), 3.25 (s, 3H, CH_3), 3.35 – 3.44 (m, OCH_2), 3.48 (s, OCH_2), 3.77 – 3.81 (m, OCH_2), 4.13 (m, OCH_2), 6.94 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 7.40 (d, 2H, C_6H_4, J_{H-H} = 8.8 Hz), 9.38 (s, 1H, SH).

A general procedure for the synthesis of clusters 7a-e and 8 is outlined below: Cluster 7a: A mixture of $[Fe_4S_4(S\text{-}tBu)_4]^{2-}\cdot 2(nBu_4N^+)$ (100 mg, 0.084 mmol) and 6a (260 mg, 0.47 mmol) in anhydrous DMF (5 ml) was stirred overnight under vacuum maintained at 100 mtorr. This allowed solvents and by-product tBuSH to be removed slowly, and a black solid was left. The residue was dissolved into 5 ml of THF, and filtered under nitrogen to remove the unreacted starting cluster, and then 10 ml of ethyl ether was added to the filtrate. The precipitate was collected by filtration, and then washed with ethyl ether (2 × 10 ml), yield: 92%. ^{1}H NMR (DMSO-d_6, ppm) δ 0.89 (t, 24H, CH_3, $J_{H\text{-}H}$ = 7.3 Hz), 1.27 (m, 16H, CH_2), 1.54 (br, 16H, CH_2), 3.12 (br, 16H, CH_2), 3.24 (br, 12H, CH_3), 3.55 (br, 8H, OCH_2), 4.14 (br, 8H, CH_2), 5.51 (br, 8H, C_6H_4), 7.53 (br, 8H, C_6H_4). ^{13}C NMR (DMSO-d_6, ppm) δ. UV-VIS (CH_2Cl_2) $\lambda_{max/nm}$ ($\varepsilon_{232} = 2.28 \times 10^4$ $cm^{-1}M^{-1}$, $\varepsilon_{286} = 1.25 \times 10^4$ $cm^{-1}M^{-1}$). Anal calcd for $C_{52}H_{80}N_2O_4S_8Fe_4$ (1337.29): C, 46.57%; H, 6.01%; N, 2.09%; S, 19.12%. Found C, %; H, %. MS (MALDI-TOF, dithranol):.

Cluster 8: yield: 90%. ^{1}H NMR (DMSO-d_6, ppm) δ 0.96 (t, 24H, CH_3, $J_{H\text{-}H}$ = 7.3 Hz), 1.23 (m, 16H, CH_2), 1.48 (br, 16H, CH_2), 3.06 (br, 16H, CH_2), 5.02 (br, 8H, C_6H_4), 7.47 (br, 8H, C_6H_4). ^{13}C NMR (DMSO-d_6, ppm) δ 14.77, 20.52, 24.30, 58.87, 112.06. UV-VIS (CH_2Cl_2) $\lambda_{max/nm}$ 232, 286 ($\varepsilon_{232} = 2.28 \times 10^4$ $cm^{-1}M^{-1}$, $\varepsilon_{286} = 1.25 \times 10^4$ $cm^{-1}M^{-1}$). An al calcd for $C_{40}H_{56}N_2O_4S_8Fe_4$ (1108.76): C, 43.33%; H, 5.09%; N, 2.53%; S, 23.13% Found C, %; H, %. MS (MALDI-TOF, dithranol):.

(5) TECHNOLOGY TRANSFER

None to date